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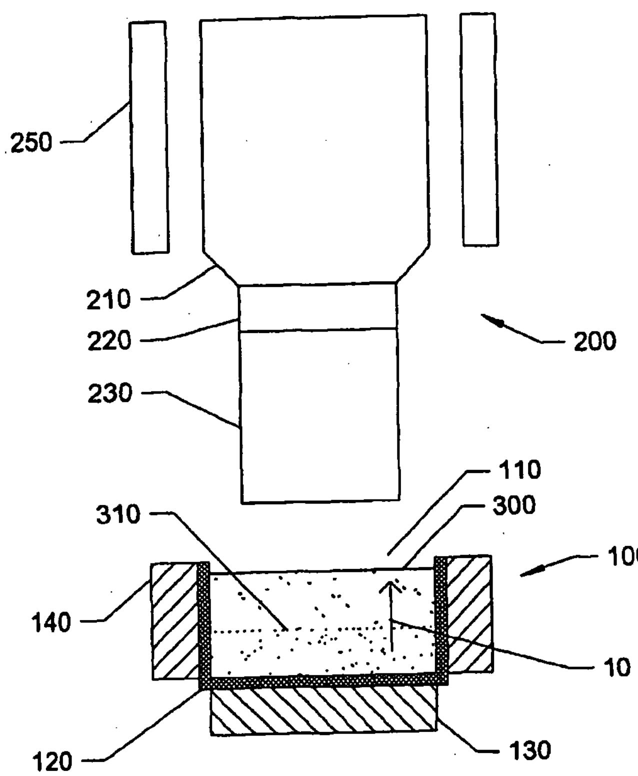
(54) Title: DIE CAST SPUTTER TARGETS



(57) Abstract: Die cast sputter targets, and methods and devices that facilitate formation of die cast sputter targets. Materials to be used in casting a sputter target are melted in a crucible (200), poured into a die (100), and allowed to cool to form the sputter target. The die utilizes heated sidewalls (140) and a cool base (130) to promote bottom to top solidification and a target having uniform top solid interfacial propagation. The die cast sputter target can be used to form layers in microelectronic devices.

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Published:
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— *with amended claims*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

DIE CAST SPUTTER TARGETS

Field of The Invention

The present invention relates, in general, to the formation of sputter targets.

Background of The Invention

Sputtering targets need to be cleaner and more cost effective to meet customer needs at smaller feature sizes. It is known that the use of finer grain targets reduces arcing when the target is being sputtered and also increases target strength (which in turn permits their use in high power applications). Sputter targets are typically used to form layers in microelectronic devices.

Sputter targets are typically made from a cast billet that is allowed to cool at a slow rate, cut and forged to refine the average grain size and add strength to the metal. Grain size is often determined using ASTM standard E112 - "Standard Test Methods for Determining Average grain size", and/or E1382 - "Standard Test Methods for Determining Average grain size Using Semiautomatic and Automatic Image Analysis".

Forging processes can be costly due to complex machining and material loss due to machining scrap generation. This is especially problematic when sputtering targets with complex shapes need to be generated using very costly high purity metals. Moreover, hot forging will not generate the required small grain sizes as hot forging often results in the targets being maintained at elevated temperatures for extended times with a corresponding increase in average grain size.

There is an ongoing need for cost effective methods to fabricate sputter targets having a very fine average grain size, which translates into a higher target mechanical strength and less potential arcing during sputtering.

Summary of the Invention

The present invention is directed to die cast sputter targets, as well as methods and devices that facilitate formation of die cast sputter targets. Materials to be used in casting a sputter target are melted in a crucible, poured into a die, and allowed to cool to form the sputter target. The die utilizes heated sidewalls and a cooled base to promote

bottom to top solidification and a target having uniform solid interfacial propagation. In some instances, the heated sidewalls may be replaced by sidewalls that simply transfer heat out of the die at a slower rate than the base. The use of such dies permits heat flow both into and out of the die to be carefully controlled. By controlling the bottom to top cooling, it is possible to avoid porosity or void generation in the part.

Casting targets into their final shape is advantageous as such targets require little, if any, machining after they are cast. Reducing or eliminating machining of targets decreases the time and expense required to produce targets and also decreases the amount of waste material produced and the amount of waste processing to be done.

Controlling heat flow both into and out of the die, such as by using a die having heated side walls, and a cooled base permits greater control over grain growth, particularly in regard to directionality of grain growth, grain size, and segregation. Quickly cooling a target being cast will help to control grain nucleation and growth and average grain size, and can help avoid segregation. High cooling/solidification rates in target production results in small grain size in the target being formed. Small grain size can be promoted as well through the use of impurity pinned suppression of grain growth during cooling.

In the case where the target has a very high aspect ratio, where the diameter is much greater than the thickness, it may not be necessary to heat the die sidewalls during solidification. For example, if a 15 inch diameter target is cast having a desired thickness of 1 inch, the side wall heat removal will only influence a region ~ 1 inch from the edge of the outer diameter during solidification. This region could experience some physical reduction in thickness, with some cavitation or porosity, but could be removed by a simple machining operation. In other cases, the material could simply be left and used, as is, since heavy sputtering does not typically occur in this region.

Modifying the rate of liquid metal introduction into cavity can be used to support more uniform cooling rates. As cooling is preferred to occur from the bottom of the target to the top of the target, there is no need to introduce all the material for the target at one time.

Die design can be used to eliminate cavities and porosity. In particular, by using reservoirs of molten metal, which are called risers. Use of a hard coating release layer such as an anodized surface facilitates removal of a target from the die after the target has solidified. Proper selection of such a coating will also facilitate heat transfer between the target being formed and the die.

Overall, using the described methods to cast high purity targets with good distribution of alloying agents should drop the cost of production of such target dramatically below the cost of using standard forging should greatly reduce the time required for target formation, and permits the formation of targets that are highly expensive and/or impossible to forge or machine. It is also contemplated that the use of such methods could greatly enhance global exact target formation capability at different manufacturing sites.

Various objects, features, aspects and advantages of the present invention will become more apparent from the following detailed description of preferred embodiments of the invention, along with the accompanying drawings in which like numerals represent like components.

Brief Description of The Drawings

The invention is best understood from the following detailed description when read in connection with the accompanying drawing. Included in the drawing are the following figures:

FIG. 1 is a side view of a crucible and a die with the die being shown in a cutaway view.

FIG. 2 is a top view of the crucible of figure 1.

FIG. 3 is a top view of the die of figure 1.

Detailed Description

The term "sputter target" as used herein includes any coil or other component that can be used to sputter and can be in any shape including cylindrical.

The term "layer" as used herein includes film and coating.

In figure 1, a casting system comprises die 100 and crucible 200. Also shown is partially formed target 300. Materials used in casting target 300 are melted in crucible 200, poured into die 100, and allowed to cool to form target 300. Heat flow into and out of die 100 is controlled through the use of heated sidewalls and a cool (preferably water cooled) base to promote bottom to top solidification and a target having uniform solid interfacial propagation. One could even use cryogenic cooling of the mold if desired.

It should be noted that in some embodiments, dies having non-heated sidewalls may be used if the rate of heat transfer out of the die cavity through the sidewalls is sufficiently lower than the rate of heat transfer through the bottom of the die.

Bottom cooled, sidewall heated non-reactive die 100 comprises die cavity 110, die lining 120, die base 130 and die sidewall 140. Die cavity 110 is preferably sized and dimensioned to allow target 300 to be cast into its final desired shape. Lining 120 is preferably a high thermal conductivity, high temperature material that does not interact with (is non-reactive to) the molten metal of target 300. A CVD diamond-like coating is the currently preferred material for use as lining 120. Die base 130 is preferably a high thermal conductivity bottom chill plate such as a copper plate coated with a sufficiently thick diamond-like coating (lining 120) on the surface. Preferred bases have a thermal conductivity greater than or equal to that of aluminum. Coatings that consist of Ta, Ta Nitride or other refractory materials can be used that have sufficient non-reactivity with the molten metal such that it can be released from the die after deposition and cooling. It is contemplated that providing for higher cooling capacity at the edges compared to the center of base 130 will help to obtain uniform directional solidification of target/material 300. Die sidewalls 140 are preferred heated, but in some instances may simply have low thermal conductivity or otherwise be adapted to transfer heat out of the die cavity at a rate less than 10% of that of the base. In some instances, cavity 110 of die/mold 100 will be cylindrically shaped. . If desired, the die base 130 can be selected out of a material such as a superalloy like Inconel™ or Hastelloy™ that does not require a liner 120. A native anodization of the material surface could suffice for die cavity without the need of

a liner. As was mentioned earlier, the die sidewall 140 would not have to necessarily be heated but is preferred for a high integrity outer edge of the cast body.

It is contemplated that it may be desirable to vary heat flow through the various portions of base 130 and sidewall 140, possibly through the use of multiple heating and cooling elements, to permit additional property control of the casting of target 300. Crucible 200 comprises high temperature reservoir 210, high temperature valve 220, spout 230, and induction coils 250. It is contemplated that any suitable source of molten material may be used to provide material to die 100. However, any such source preferably comprises a mechanism for controlling the flow rate of material into die 100 so the flow rate can be controlled to promote uniform cooling of target 300. Spout 230 could also have a multiplicity of openings, like a showerhead, above the die 100 to ensure rapid and even delivery of molten metal into the die cavity, where it will rapidly solidify. It is also contemplated that melting the molten material may be accomplished in a number of ways, including, but not necessarily limited to the use of induction coils and e-beams.

Target 300 is formed and contained in die 100. As shown in figure 1, the material of target 300 is solid below interface 310 and molten above interface 310. It is contemplated that the composition of the material selected in target 300, will vary depending on the particular application(s) contemplated for target 300. Preferred materials at this time include Al, Al alloys and Cu. In the future, higher temperature metal systems, like Ti and Zr, could be manufactured using such an approach. In addition, it is advantageous to introduce small amounts of alloying agents to the molten metal to enhance nucleation and growth properties of the formed solid target material. However, it is contemplated that the introduction of impurities into the chosen material may promote impurity pinned suppression of grain growth during cooling which is desirable for sputtering target properties. This approach can help to retain fine average grain sizes in the process of solidification.

Arrow 10 shows the direction of solidification of material/target 300. Heat flow through the target during casting is principally in a direction opposite of the direction of solidification. Although heat loss in a typical die would occur through both the sides and

bottom of the die, the heated sidewalls and cooled bottom plate of die 100 permit heat flow and cooling to occur in a more linear fashion with a corresponding bottom to top direction of grain growth. If the sidewalls aren't heated, the use of a cooled bottom plate and insulated sidewalls can also ensure fast vertical solidification and a dense target. In any instances, it is contemplated that cooling the bottom of the die to 70%, or more preferably to 20% of the melting point of the melting point of the molten material being formed into a target will facilitate the desired grain growth.

A method of using the casting system of figure 1 may comprise the following steps. Place high purity material in a high temperature melting vessel/crucible 200 under vacuum or inert gas in order to avoid the formation of oxides. Melt the metal/material with an induction or e-beam source such as induction coils 250. Set up a die 100 having heated sidewalls 140 and a high conductivity bottom chill plate/base 130, and having all inner surfaces coated with a high conductivity, non-reactive high temperature material as lining 120 which does not interact with the molten metal. The molten metal is deposited into the die cavity 110, from the melting vessel 200, where it rapidly solidifies from bottom to top with a uniform top solid interfacial propagation. The die cast material/target 300 is then released from the mold and finished if desired/needed.

Targets formed using the described methods and devices will have small, well controlled average grain sizes which will provide low arcing during operation.

The described devices and methods facilitate carefully controlled casting by permitting the temperatures of the sidewalls and base of the die to be modified over time to control the solidification and average grain size. Obtainable average grain sizes are preferred to be less than 500 µm after solidification, more preferably less than 100 µm after solidification, and still more preferably less than 50 µm after solidification. Grain sizes as given herein are determined in accordance with ASTM standards E112 and/or E1382.

Thus, specific embodiments and applications of devices and formation methods for the generation of cast sputtering targets have been disclosed. It should be apparent, however, to those skilled in the art that many more modifications besides those already described are possible without departing from the inventive concepts herein. The

inventive subject matter, therefore, is not to be restricted except in the spirit of the appended claims. Moreover, in interpreting both the specification and the claims, all terms should be interpreted in the broadest possible manner consistent with the context. In particular, the terms "comprises" and "comprising" should be interpreted as referring to elements, components, or steps in a non-exclusive manner, indicating that the referenced elements, components, or steps may be present, or utilized, or combined with other elements, components, or steps that are not expressly referenced.

CLAIMS

What is claimed is:

1. A die cast sputter target formed from a casting of liquid metal that has an average grain size below 500um after solidification.

2. The sputter target of claim 1 wherein the target has an average grain size below 50um after solidification.

3. The sputter target of claim 1 comprising one or more of Al, Cu, Ti or Zr.

4. A layer formed from said target of claim 1.

5. A microelectronic device comprising said layer of claim 3.

6. A method of casting a sputter target comprising:
providing a die and a high temperature melting vessel;
placing a high purity casting material in the melting vessel;
melting the casting material;
transferring the casting material into a cavity of the die;
simultaneously transferring heat into and out of the casting material while allowing it to solidify into a target in the die;
releasing the target from the die.

7. The method of claim 6 wherein the material is melted via an induction or e-beam source under vacuum or inert gas.

8. The method of claim 6 wherein the die has a base adapted to transfer heat out of the casting material as it solidifies.

9. The method of claim 8 wherein the die has at least one heated sidewall adapted to transfer heat into the casting material as it solidifies.

10. A sputter target casting die comprising:
a cavity adapted to receive a quantity of casting material;

a base adapted to transfer thermal energy out of any material contained in the cavity;

at least one sidewall adapted to transfer thermal energy into any material contained in the cavity.

11. The die of claim 10 wherein the cavity is lined with a substantially non-reactive material having a high coefficient of thermal conductivity.

12. The die of claim 10 wherein the base has a thermal conductivity greater than or equal to that of aluminum.

13. The die of claim 10 wherein the at least one sidewall is adapted to transfer thermal energy into or out of any material contained in the cavity at a rate of less than 10% of that of the base.

14. The die of claim 10 wherein the base is adapted to be maintained at a temperature equal to or less than 70% of the melting point of any molten casting material.

15. The die of claim 14 wherein the base is adapted to be maintained at a temperature equal to or less than 20% of the melting point of any molten casting material.

16. A casting system comprising:

a quantity of casting material;

a high temperature melting vessel adapted to contain the casting material and to permit the casting material to flow out of the vessel at a controlled rate;

a die adapted to receive casting material flowing out of the high temperature melting vessel, the die having at least one sidewall adapted to transfer thermal energy into any casting material contained in the die, and a base adapted to transfer thermal energy out of any casting material contained in the die.

17. The system of claim 16 wherein the die comprises a cavity lined with a high thermal conductivity, high temperature material that is substantially non-reactive to the casting material.

18. The system of claim 17 wherein the high temperature melting vessel comprise an induction or e-beam source for transferring thermal energy into any casting material contained in the vessel, and a high temperature valve adapted to control the flow of any material between the vessel and the die.

AMENDED CLAIMS

[received by the International Bureau on 19 May 2003 (19.05.03)

CLAIMS

1. A die cast sputter target having a uniform solid interfacial propagation formed from a casting of liquid metal that has an average grain size below 500um after solidification.

2. The sputter target of claim 1 wherein the target has an average grain size below 50um after solidification.

3. The sputter target of claim 1 comprising one or more of Al, Cu, Ti or Zr.

4. A layer formed from said target of claim 1.

5. A microelectronic device comprising said layer of claim 3.

6. A method of casting a sputter target comprising:

   providing a die and a high temperature melting vessel;

   placing a high purity casting material in the melting vessel;

   melting the casting material;

   transferring the casting material into a cavity of the die;

   simultaneously transferring heat into and out of the casting material while allowing it to solidify into a target in the die; and

   releasing the target from the die.

7. The method of claim 6 wherein the material is melted via an induction or e-beam source under vacuum or inert gas.

8. The method of claim 6 wherein the die has a base adapted to transfer heat out of the casting material as it solidifies.

9. The method of claim 8 wherein the die has at least one heated sidewall adapted to transfer heat into the casting material as it solidifies.

10. A sputter target casting die comprising:

a cavity adapted to receive a quantity of casting material;

a base adapted to transfer thermal energy out of any material contained in the cavity; and

at least one sidewall adapted to transfer thermal energy into any material contained in the cavity.

11. The die of claim 10 wherein the cavity is lined with a substantially non-reactive material having a high coefficient of thermal conductivity.

12. The die of claim 10 wherein the base has a thermal conductivity greater than or equal to that of aluminum.

13. The die of claim 10 wherein the at least one sidewall is adapted to transfer thermal energy into or out of any material contained in the cavity at a rate of less than 10% of that of the base.

14. The die of claim 10 wherein the base is adapted to be maintained at a temperature equal to or less than 70% of the melting point of any molten casting material.

15. The die of claim 14 wherein the base is adapted to be maintained at a temperature equal to or less than 20% of the melting point of any molten casting material.

16. A casting system comprising:

a quantity of casting material;

a high temperature melting vessel adapted to contain the casting material and to permit the casting material to flow out of the vessel at a controlled rate; and

a die adapted to receive casting material flowing out of the high temperature melting vessel, the die having at least one sidewall adapted to transfer thermal energy into any casting material contained in the die, and a base adapted to transfer thermal energy out of any casting material contained in the die.

17. The system of claim 16 wherein the die comprises a cavity lined with a high thermal conductivity, high temperature material that is substantially non-reactive to the casting material.

18. The system of claim 17 wherein the high temperature melting vessel comprise an induction or e-beam source for transferring thermal energy into any casting material contained in the vessel, and a high temperature valve adapted to control the flow of any material between the vessel and the die.

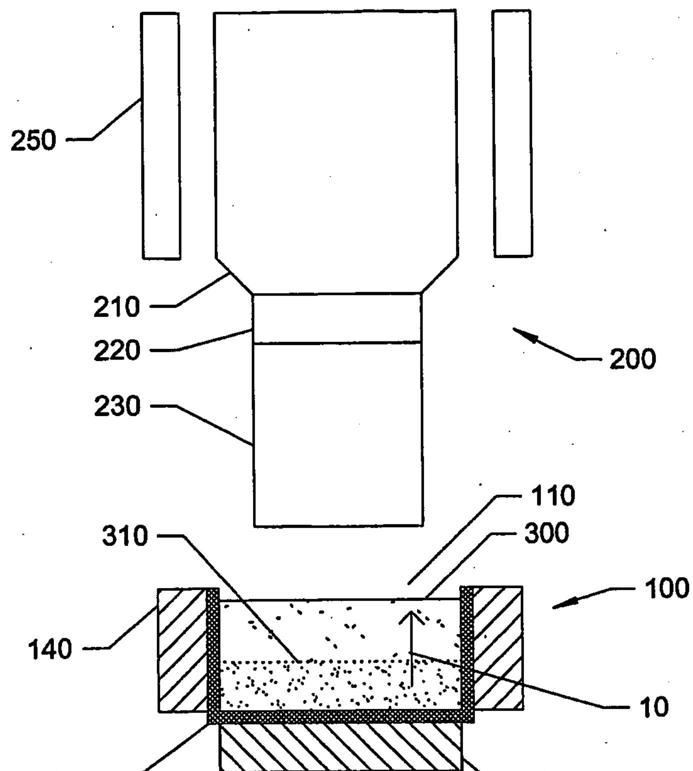

Fig. 1

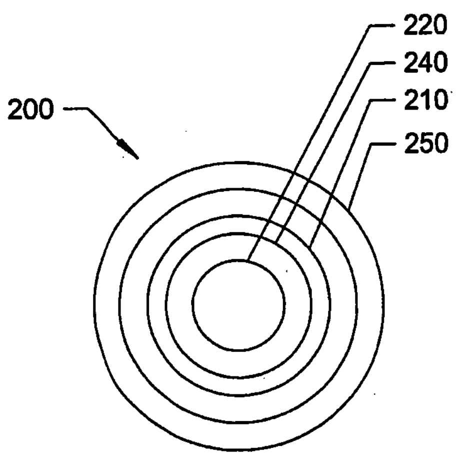

Fig. 2

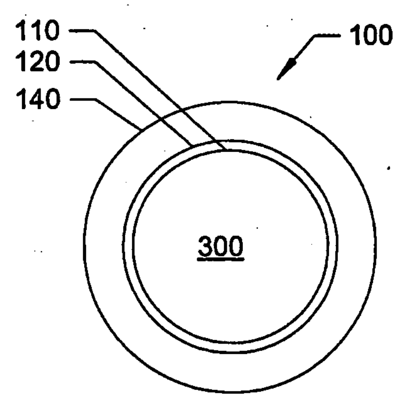

Fig. 3

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US02/36069

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) : B22D 27/09, 41/005, 17/00; C22C 21/00, 9/00, 14/00, 16/00
US CL : 164/348,113,125,312; 420/417,422,469,528
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 164/348,113,125,312; 420/417,422,469,528

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EAST

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 6,368,427 B1 (SIGWORTH) 09 April 2002, abstract, col. 6 line 37.	1-5
X --- Y	US 4,834,166 A (NAKANO) 30 May 1989, col. 3 lines 6-7, 30-35, col. 7 lines 10-11, col. 4 lines 40-69.	6, 8-18 --- 7

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:
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"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
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"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
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Date of the actual completion of the international search	Date of mailing of the international search report
03 MARCH 2003	20 MAR 2003

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